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Indian Standard

METHODS FOR DETERMINATION OF pH VALUE OF AQUEOUS EXTRACTS OF TEXTILE MATERIALS (*First Revision*)

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NEW DELHI 110002

Indian Standard

METHODS FOR DETERMINATION OF pH VALUE OF AQUEOUS EXTRACTS OF TEXTILE MATERIALS (First Revision)

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Indian Standard
METHODS FOR
DETERMINATION OF pH VALUE OF
AQUEOUS EXTRACTS OF TEXTILE MATERIALS
(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 31 October 1983, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

0.2 This standard first published in 1961, has been revised to align it with ISO 3071-1980 'Textiles — Determination of pH of the aqueous extracts' issued by the International Organization for Standardization.

0.3 The pH of aqueous extract of the textiles affords a useful index to its processing history. In addition, it is becoming more common to demand that the textile, in its various forms, shall conform to certain limits in respect of its acidity or alkalinity, often expressed in terms of pH values of aqueous extracts. The values obtained for the pH of the aqueous extract of samples of textiles, by the methods specified in this standard, should not be used to give quantitative estimate of the acidity or alkalinity of the textile. Such an interpretation may be misleading, particularly for pH values less than 3 or greater than 11.

1. SCOPE

1.1 This standard prescribes methods for determination of pH value of aqueous extracts of textiles. The methods are applicable to textile in any form (fibres, yarns or fabrics) provided, that a small representative sample may be obtained which is in or may be reduced to, a form which permits a ready exchange of liquid between the interior of the material and the water used in preparing the extract.

1.2 Two methods of preparation of aqueous extracts have been prescribed in this standard. For kier-boiled or scoured and bleached textile materials which have not been finished, pH values of aqueous

extracts prepared by the cold method and by the hot method would be the same. Hence any one of the methods may be followed. However, for finished or proofed fabrics the two values may differ depending upon the nature of finish or proofing of the fabrics; in such cases pH values of cold extract as well as hot extract should be separately determined.

2. PRINCIPLE

2.1 A specimen of the material under test is extracted with distilled or deionized water and the pH of the aqueous extract is determined electrometrically with the use of glass electrodes.

3. SAMPLING

3.1 Sample to determine the conformity of a lot to a specification shall be selected so as to be representative of the lot.

3.2 Sample drawn in compliance with an agreement between the buyer and the seller to evaluate pH value of the aqueous extract of the textile material shall be held to be representative of the lot.

4. PREPARATION OF TEST SPECIMENS

4.1 Take a laboratory sample representative of the bulk of the material and sufficient to provide all the test specimens required. Cut the laboratory test sample into pieces approximately 5 mm size or of dimensions such that the test samples will rapidly wet out. Manipulate the material as little as possible.

5. CONDITIONING OF TEST SAMPLE

5.1 Prior to test, the test sample shall be conditioned to moisture equilibrium in a standard atmosphere at 65 ± 2 percent relative humidity and $27 \pm 2^\circ\text{C}$ temperature (see IS : 6359-1971*).

6. APPARATUS

6.1 Glass Stoppered Flasks — made of chemically resistant glass for preparation of the aqueous extract.

6.2 Mechanical Shaker — providing a to and fro movement at a rate of 60 per minute or a rotational frequency of 30 per minute.

6.3 pH Meter — graduated in 0.1 unit of pH and with suitable electrode system.

6.4 Beakers — made of chemically resistant glass of capacity 250 ml.

6.5 Balance — accurate to 0.05 g.

*Method for conditioning of textiles.

6.6 Erlenmeyer Flask — made of chemically resistant glass and of 250 ml capacity fitted with a glass stopper.

6.7 Reflux Condenser

7. REAGENTS

7.1 Distilled or Deionized Water — having a pH between 6.5 and 7.0 and maximum conductivity of 2×10^{-6} S/cm* at $27 \pm 2^\circ\text{C}$.

7.2 Buffer Solutions — of pH similar to that being determined, for standardization of pH meter before measurement. The solutions recommended are given in 7.2.1 and 7.2.2.

7.2.1 Primary Standard — potassium hydrogen phthalate ($\text{HOOC.C}_6\text{H}_4\text{COOK}$), 0.05 mol/l, pH 4.00 at 15°C , 4.001 at 20°C , 4.005 at 25°C and 4.011 at 30°C .

7.2.2 Secondary Standard — disodium tetraborate decahydrate, ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) 0.05 mol/l, pH 9.28 at 10°C , 9.21 at 20°C , 9.13 at 30°C and 9.06 at 40°C .

8. PROCEDURE

8.1 Preparation of the Aqueous Extract

8.1.1 Cold Method — Prepare the extract in triplicate at the temperature of laboratory (which shall be recorded) by taking a test specimen weighing 2 ± 0.05 g and 100 ml of the distilled or deionized water (see 7.1) into a stoppered glass flask (6.1). Agitate the flask for 20-30 min by hand to ensure that the specimen is properly wetted out, then shake it mechanically for 20-30 minutes (see 6.2).

8.1.2 Hot Method — Rinse the Erlenmeyer flask with distilled or deionized water. Take one test specimen weighing 2 ± 0.05 g and add to this 100 ml of distilled or deionized water. Boil the contents for one hour under reflux condenser. Stopper the flask and cool the contents to room temperature.

8.2 Measurement of pH of Aqueous Extract — Follow the procedure specified in either 8.2.1 or 8.2.2. If electrode systems other than those specified below are used, take similar precautions. Carry out each test at the same temperature which should be near the ambient temperature, avoiding any increase in temperature greater than 5°C .

8.2.1 Procedure Using a Morton Cell Type Electrode System

8.2.1.1 Standardize the meter at the temperature of the extract to be measured (see 7.2).

*Siemens per centimetre.

8.2.1.2 Wash the cell several times with distilled water until the indicated pH no longer changes. This requires a considerable volume of distilled water.

8.2.1.3 Pour into the cell sufficient of the first extract to cover the bulb of the glass electrode. Re-stopper the flask. Allow the cell to stand for 3 min. Read the pH value. Drain the cell and pour in a new portion of the same extract. Re-stopper the flask. Allow the cell to stand for 1 min and read the pH. Repeat these operations until the indicated pH reaches its extreme steady value. Discard the first extract.

8.2.1.4 Without washing out the cell, pour in a sufficient quantity of the second extract to cover the bulb of the glass electrode. Read the pH immediately. Drain the cell and introduce a new portion of the extract. Read the pH value again. Repeat these operations until the indicated pH attains its extreme steady value.

8.2.1.5 Record this value to the nearest 0.1 unit of pH. Discard the extract.

NOTE — For an alkaline extract (pH value greater than 7), the highest steady pH indicated is recorded as the pH value of the extract and for acid extract (pH value less than 7) the lowest steady pH indicated is recorded as the pH value of the extract. This is referred to as the 'extreme steady value'.

8.2.1.6 Determine the pH of the third extract using the above procedure without rinsing the cell.

8.2.1.7 The pH values of the second and third extracts are recorded as duplicate determination.

8.2.2 *Procedure Using a Dipping Electrode System*

8.2.2.1 Standardize the meter at the temperature of the extract to be measured (see 7.2).

8.2.2.2 Wash the electrodes until the indicated pH value changes by not more than 0.05 in 5 min. If this cannot be realised, replace the glass and/or reference electrodes.

8.2.2.3 Decant the first extract, with the exclusion of the textile material, into a beaker (6.4). Immediately immerse the electrodes to a depth of at least 1 cm and stir very gently with a glass rod until the pH attains its extreme steady value (see Note under 8.2.1.5).

8.2.2.4 Decant the second extract into a beaker. Transfer the electrodes, without washing, into the second beaker, lowering them gently to a depth of at least 1 cm and allow to stand without stirring until the pH attains its extreme steady value. Record this value to the nearest 0.1 unit of pH (see Note under 8.2.1.5).

8.2.2.5 Decant the third extract in another beaker and transfer the electrodes to this beaker, again without washing. Determine the pH value as described above.

8.2.2.6 The pH values of the second and third extracts are recorded as duplicate determinations.

9. CALCULATION AND EXPRESSION OF RESULTS

9.1 Give the values obtained for the second and third extracts as 'first and second measurements'.

9.2 Calculate their mean to the nearest 0.05 unit of pH .

10. INDEX OF DIFFERENCE

10.1 If the value of the pH measured is less than 3 or greater than 9 determine the index of difference as follows:

- a) Introduce 10 ml of the prepared aqueous extract into a beaker (6.4) and add 90 ml of distilled or deionized water (7.1).
- b) Measure the pH of the solution to 0.1 unit of pH following the procedures specified in 8.2.1 or 8.2.2.
- c) The difference between the pH of the aqueous extract prepared as in 8.1 and that of the dilution to 1/10 is the index of difference.

10.2 This index of difference, which should never be greater than unity, is especially high when the textile contains strong acids or strong bases and these are not buffered by weak acids or weak bases.

11. TEST REPORT

The test report shall include the following information:

- a) Reference to this Indian Standard;
- b) Hot method or cold method;
- c) Type of electrodes used;
- d) The pH of the distilled water used;
- e) The temperature of the laboratory;
- f) The results obtained, expressed in the form indicated in 9;
- g) If necessary, the index of difference (see 10); and
- h) Any factor likely to have an effect on the results, including any resistance to wetting out of the specimen.

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